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(54) PERFUME CARRIERS BASED ON POLYURETHANE- POLYUREA GELS

(71) We, BAYER AKTIENGESELLSCHAFT, a body corporate organised under the laws of Germany, of 509 Leverkusen, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to perfume carriers based on polyurethane - polyurea gels; more particularly, it relates to new perfume carriers based on stable aqueous and/or alcoholic polyurethane - polyurea gels.

The principle of using gels as perfume carriers is known and the gels previously used were all based on natural products, in particular on polysaccharides and proteins. Gels based on carrageenates, alginates, pectins or gelatin are examples.

These previously known products have decided disadvantages. As a general rule, reproducible production of gels from natural substances is possible only if very pure starting materials are used. This means that only high quality products which are generally obtained by expensive and complicated purification processes may be employed. Moreover, the raw materials are often not available in sufficient quantities.

Another disadvantage of natural products is that there is little scope for variation of their properties. Although gels produced from the two classes of natural substances, polysaccharides and proteins, differ slightly in their properties, it is difficult to adjust their properties optimally to the perfume substance used in any given case, since perfume sub-

stances may have completely different chemical structures, for example, they may have a terpene structure or they may contain ester, ether or ketone functions. It is therefore desirable to have gels which may be optimally adapted to the perfume substances in their structure so that maximum liberation of the scent uniformly distributed over a period of time will be achieved.

Another disadvantage of gels based on natural substances is their shrinkage which is not proportional to the external dimensions. Although this shrinkage does not directly affect the liberation of scent, it is visually very displeasing and therefore undesirable. Particularly unattractive shrinking occurs when perfume carriers based on gels are left uncovered and exposed to air.

Another disadvantage is that gels based on natural substances have no dimensional stability to heat. They begin to flow at temperatures which may be as low as 40°C, depending on the nature of the gel matrix and the nature and composition of the gel. This is, of course, particularly unpleasant when the atmospheric temperature is high, as in tropical and sub-tropical climates. In extreme cases, the perfume carrier then collapses to a pasty mass.

It has now been found that gel-based perfume carriers which are not affected by the temperature and may easily be varied in their properties may be obtained by using polyurethane - polyurea polymers of the type which have partly been disclosed in German OS No. 2,347,299 as gel-forming agents.

The gels obtained in this way do not have

the disadvantages mentioned above which are characteristic of the previously known gel-forming agents.

Being synthetic products, they are inexpensive and always available in the same, reproducible high quality. Their gel properties may be optimally adapted to the requirements of any individual perfume by changing their structure, particularly by varying the nature and quantity of the polyol and of the isocyanate. These gels undergo shrinkage which is proportional to their external dimensions. Their type of cross-linking, which is due to their main valency forces, also makes them highly dimensionally stable to heat. They undergo no flow even at temperatures up to 100°C.

These perfume carriers which are temperature resistant and easily variable in their properties may be obtained by reacting isocyanate prepolymers based on polyethers which contain ethylene oxide units with diamines or polyamines or water as chain-lengthening agents in water and/or alcohols in the presence of perfume substances and, if required, other additives. The term "gel" used for such perfume carriers is intended to denote the physical nature of the gel-like end product rather than the exact polymer physical structure in accordance with present day views of the colloid chemistry of this state.

The reaction of the individual components to form the gels takes place spontaneously. Owing to the high speed of formation, the process of producing the perfume carriers may easily be rendered continuous, which is highly advantageous for many purposes.

The present invention therefore relates to perfume carriers based on polyurethane - polyurea gels and to a process for the preparation thereof, which is characterised in that isocyanate prepolymers of polyethers which contain at least 40% by weight, ethylene oxide units are reacted with diamines or polyamines or water as chain-lengthening agents in water and/or alcohols in the presence of perfume substances and, if required other additives.

Either water or alcohols or mixtures of the two may be used as dispersing agents for gel formation so that the gels may be divided into hydrogels and alcohol gels. The alcohols used as dispersing agents may be either highly volatile monohydric alcohols, such as ethanol, isopropanol or butanol, or they may be comparatively non-volatile polyhydric alcohols, such as ethylene glycol, diethylene glycol, glycerol and trimethylol propane.

Preparation of the perfume carriers may be carried out by various methods. If desired, all the components may be added together simultaneously, i.e. the prepolymer, the dispersing agent (water and/or alcohol), the perfume substance, the chain-lengthening agent and, if required, other additives, but the process is preferably carried out step-wise. In that case,

the perfume substance, alone or mixed with other additives, such as dyes, preservatives and, if required, cross-linking agents, such as amines, is dissolved or emulsified in the dispersing agent. At the same time, an emulsion or solution of the prepolymer in the dispersing agent is prepared and the two solutions or emulsions are mixed together in substantially stoichiometric proportions. The mixture is then poured into moulds in which the reaction to form the gel perfume carriers is completed within a few seconds or minutes.

The end product may have the consistency of a compact solid or of a foam, depending on the chain-lengthening agent used. Compact gels are obtained when amines are used as chain-lengthening agents, while foamy products are obtained when water is used on account of the reaction of the isocyanate groups with water to form gaseous carbon dioxide.

Vigorous mixing of the starting components facilitates preparation of the perfume carriers and improves the quality of the gels. In the simplest case, mixing may be carried out in a zone of increased turbulence produced by mechanical stirrers, but better results are obtained using high speed mixing apparatus, such as impeller homogenisers or stirrer mixing chambers of the type known in the literature and used in commercial polyurethane foaming machines. Vigorous mixing may also be achieved by using the mixing devices of polyurethane foaming machines in which mixing is carried out by counterflow injection.

The prepolymers which are obtained from polyethers containing at least 40%, by weight, ethylene oxide units and polyisocyanates and which still contain free isocyanate end groups are prepared in known manner by reacting the corresponding polyethers with an excess quantity of polyisocyanate, in other words using an NCO/OH ratio greater than 1, preferably from 1.5 to 20, more particularly from 2 to 10.

The optimum ratio for carrying out the process depends on the molecular weight and the functionality and structure of the polyether. The isocyanate contents of the prepolymers should generally be between 2 and 20%, by weight, preferably between 5 and 10%, by weight.

If amines are used as chain-lengthening agents, it is generally required to use substantially stoichiometric quantities of prepolymers and amines for formation of the perfume carrier gels, but it has been found that when the process is carried out on a technical scale it is advantageous to use an excess of amine component, i.e. an NH_2/NCO ratio greater than 1, preferably from 1 to 1.2, in order to obtain a sufficient residence time in the mixing apparatus.

If water is used as chain-lengthening agent, the isocyanate groups and water may also be used in stoichiometrically equivalent quantities,

but, if desired, and especially for producing foamy gels, a substantially larger quantity of water than would be required for the reaction with isocyanate groups may be used. In that case, it is particularly advantageous also to use water as the dispersing agent, so that foamy hydrogels are obtained. The water then serves a double function, as reactant for the isocyanate and as dispersing agent.

The quantity of water and/or alcohol present during formation of the gels is not critical and may vary within wide limits. Based on the total quantity of gel, the weight of alcohol and/or water may be up to 98%, but the properties of the gels obtained are strongly influenced by the ratio of polymer to dispersing agent. The gels are generally increasingly more stable and harder with increasing polymer content and progressively softer and structurally less stable with decreasing polymer content down to a lower limit of about 2%, by weight.

It is particularly surprising that the gel-based perfume carriers according to the present invention are exceptionally stable. No visible phase separation which would manifest itself, for example, by cloudiness of the material occurs even after prolonged storage. The dimensional stability is preserved even if the gels are stored at elevated temperatures and again no phase separation may be observed under these conditions. The dispersing agent is very firmly bound in the gel, but may escape more or less rapidly, depending on its vapour pressure, so that the gel gradually shrinks in proportion to its external dimensions. In the course of this process, the perfume substance diffuses from the gel and evaporates into the surroundings.

The polyethers used as one of the starting materials for the preparation of the perfume carriers according to the present invention generally contain at least two active hydrogen atoms and generally have a molecular weight of from 500 to 10,000, preferably from 2000 to 8000 and contain at least 40%, by weight, preferably more than 50%, by weight, of ethylene oxide groups. These polyethers may be prepared by the reaction of compounds which contain reactive hydrogen atoms, e.g. polyhydric alcohols, with ethylene oxide and, if desired, also other alkylene oxides, such as propylene oxide, butylene oxide, styrene oxide or epichlorohydrin or mixtures of these alkylene oxides.

Suitable polyhydric alcohols and phenols include, for example, ethylene glycol, diethylene glycol, higher polyethylene glycols, propane - 1,2 - diol, propane - 1,3 - diol, butane - 1,4 - diol, hexane - 1,6 - diol, decane - 1,2 - diol, butene - 2 - diol - (1,4), glycerol, butane - 2,4 - diol, hexane - 1,3,6 - triol, trimethylolpropane, resorcinol, hydroquinone, 4,6 - di - tert. - butyl pyrocatechol, 3 - hydroxy - 2 - naphthol, 6,7 - dihydroxy -

1 - naphthol, 2,5 - dihydroxy - 1 - naphthol, 2,2 - bis - (p - hydroxyphenyl) - propane, bis - (p - hydroxyphenyl) - methane and α,α,ω - tris - (hydroxyphenyl) - alkanes, such as 1,1,2 - tris - (hydroxyphenyl) - ethane or 1,1,3 - tris - (hydroxyphenyl) - propane.

Other suitable polyethers include the 1,2 - alkylene oxide derivatives of ammonia or of aliphatic or aromatic monoamines or polyamines, such as methylamine, ethylene diamine, N,N - dimethyl ethylene diamine, tetra- or hexa - methylene diamine, diethyl triamine, ethanolamine, diethanolamine, oleyldiethanolamine, methyldiethanolamine, triethanolamine, aminoethylpiperazine, *o*-, *m*- and *p*-phenylenediamine, 2,4- and 2,6 - diamino-toluene, 2,6 - diamino - *p* - xylene and multi-nuclear and condensed aromatic polyamines, such as naphthylene - 1,4 - diamine, naphthylene - 1,5 - diamine, benzidine, toluidine, 2,2' - dichloro - 4,4' - diamino-diphenylmethane, 1 - fluoreneamine, 1,4 - anthradiamine, 9,10 - diaminophenanthrene or 4,4' - diaminoazobenzene. Resin - forming materials, such as phenols and resols, may also be used as starting molecules for the formation of the polyethers.

All these polyethers are synthesised with the use of ethylene oxide and, optionally, other 1,2 - alkylene oxides.

The starting components used according to the present invention also include aliphatic, cycloaliphatic, araliphatic, aromatic and heterocyclic polyisocyanates of the type which have been described, for example, by W. Siefken in Justus Liebigs Annalen der Chemie, 562, pages 75 to 136, for example, ethylene diisocyanate, tetramethylene - 1,4 - diisocyanate, hexamethylene - 1,6 - diisocyanate, dodecane - 1,12 - diisocyanate, cyclobutane - 1,3 - diisocyanate, cyclohexane - 1,3- and 1,4 - diisocyanate and mixtures of these isomers, 1 - isocyanato - 3,3,5 - trimethyl - 5 - isocyanato methylcyclohexane (German Auslegeschrift No. 1,202,785), hexahydrotolylene - 2,4- and 2,6 - diisocyanate and mixtures of these isomers, hexahydrophenylene - 1,3- and/or -1,4 - diisocyanate, perhydrodiphenylmethane - 2,4'- and/or 4,4' - diisocyanate, phenylene - 1,3- and -1,4 - diisocyanate, tolylene - 2,4- and -2,6 - diisocyanate and mixtures of these isomers, diphenylmethane - 2,4'- and/or 4,4' - diisocyanate, naphthylene - 1,5 - diisocyanate, triphenylmethane - 4,4',4'' - triisocyanate, polyphenol - polymethylene - polyisocyanates which may be obtained by aniline - formaldehyde condensation followed by phosgenation and which have been described, for example, in British Patent Nos. 874,430 and 848,671, perchlorinated aryl polyisocyanates which have been described, for example, in German Auslegeschrift No. 1,157,601, polyisocyanates which contain carbodiimide groups as described in German Patent No. 1,092,007, the diisocyanates described in U.S. Patent

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No. 3,492,330, polyisocyanates containing allophanate groups according to British Patent No. 994,890, Belgian Patent No. 761,626 and published Dutch Patent Application No. 7,102,524, polyisocyanates containing isocyanurate groups as described, for example, in German Patent Nos. 1,022,789; 1,222,067 and 1,027,394 and in German Offenlegungsschrift Nos. 1,929,034 and 2,004,048 polyisocyanates which contain urethane groups as described, for example, in Belgian Patent No. 752,261, or in U.S. Patent No. 3,394,164, polyisocyanates containing acylated urea groups according to German Patent No. 1,230,778, polyisocyanates containing biuret groups as described, for example, in German Patent No. 1,101,394, in British Patent No. 889,050 and in French Patent No. 7,017,514 polyisocyanates prepared by telomerisation reactions as described, for example, in Belgian Patent No. 723,640, polyisocyanates containing ester groups, e.g. those mentioned in British Patent Nos. 956,474 and 1,072,956, in U.S. Patent No. 3,567,763 and in German Patent No. 1,231,688, or reaction products of the above-mentioned isocyanates with acetals according to German Patent No. 1,072,385.

Aliphatic, cycloaliphatic or aromatic diamines or polyamines are used as low molecular weight cross-linking or chain-lengthening agents. The following are examples of such compounds: ethylene diamine, hexamethylene diamine, diethylene triamine, hydrazine, guanidine carbonate, N,N' - bis - (3 - aminopropyl) - ethylenediamine, N,N' - bis - (2 - aminoethyl) - ethylene diamine, 4,4' - dimethylamino - diphenylmethane, 4,4' - dimethylamino - 3,3' - dimethyl - diphenylmethane, 4,4' - diamino - diphenylmethane and 2,4- and 2,6 - diaminotoluene.

The perfume substances which may be used according to the present invention for the preparation of the perfume carriers include all the aromatic individual components or compositions known under the headings of ethereal oils, perfumes or scents. It is immaterial to the process according to the present invention whether the perfume substances are soluble in the dispersing agent or may only be emulsified or dispersed therein.

The following are examples of ethereal oils: aniseed oil, oil of bergamot, camphor oil, citronella oil, lemon oil, eucalyptus oil, the various pine needle oils, geranium oil, oil of lavender, lemon grass oil, clove oil, oil of orange, peppermint oil, attar of roses, spike lavender oil, oil of turpentine and oil of cinnamon.

These are complex mixtures of alcohols, aldehydes, ketones, esters, oxides, lactones, terpenes and many other, in some cases as yet unidentified compounds.

The term "scents" is used here to denote chemically exactly defined individual sub-

stances which may either be isolated from ethereal oils or produced synthetically, for example anethole, anisaldehyde, vanillin and citronellal. By "perfumes" are meant aromatic mixtures in a solvent, and these mixtures may be composed of ethereal oils and scents.

Preparation of the perfume carriers may be carried out with the addition of considerable quantities (up to about 50 volumes percent) of various fillers, such as silicates, various types of silica - aluminium oxides, tin oxides, antimony trioxides, titanium dioxide, graphite and graphitised carbon, carbon black, retort carbon, drift sand, various cement powders and various inorganic and organic dye pigments, such as iron oxide pigments, lead chromate, lead oxide and red lead. Short or long fibres of natural or synthetic materials, such as cellulose powder, may also be used as fillers. The use of fillers has the advantage of prolonging the aroma of the perfumed gels.

Preparation of the perfume carriers may also include the addition of preservatives to prevent bacterial and mould growths or the addition of surface active agents or solutions of other natural or synthetic polymers.

If desired, a gaseous component may be added during formation of the gel to produce perfume carriers in the form of foams which have a substantially lower gross density (kg/m³) than the compact material, depending on the quantity of gas incorporated.

One particular advantage of the process according to the present invention is that it may very easily be rendered continuous.

For this purpose, the prepolymers containing isocyanate groups and, separately from these prepolymers, the chain-lengthening agent and dispersing agent are continuously introduced into a mixing zone at a temperature above the melting point, but below the decomposition point of the isocyanate prepolymer and of the chain-lengthening agent and at such a rate that the isocyanate polyaddition reaction is not entirely completed in the mixing zone. The polyurethane gel which is still in a fluid or deformable state and contains a high proportion of water or alcohol is then continuously removed from the mixing zone and, if desired, it is subsequently passed through a short reaction tube which is fitted with a shaped mouthpiece so that the perfume carrier consisting of polyurethane gel is obtained as a profiled endless strand, band or sheet.

The perfume carriers obtained in this way may be used for various purposes where improvement of the atmosphere is desired, both for domestic use and for industry.

Example 1

(a) Preparation of the prepolymer:

687 parts, by weight, of a polyether (hydroxyl number 28) based on glycerol and obtained from 60%, by weight, of ethylene oxide and 40%, by weight, of propylene oxide

- are heated to 120°C for 30 minutes, with stirring, in a reaction vessel together with 113 parts, by weight, of tolylene diisocyanate (80% of 2,4- and 20% of 2,6-isomer). Stirring of the reaction mixture is then continued for 2 hours at this temperature. The prepolymer obtained has an isocyanate content of 4.6%, by weight, and a viscosity of 6300 centipoises at 25°C.
- (b) Preparation of the perfume carrier:
16.5 parts, by weight, of the prepolymer described under 1(a) are added within 5 seconds with stirring (speed of stirrer 1200 revs/min.) to a solution of:
- 115 parts, by weight, of water,
10.5 parts, by weight, of perfume oil (mixture of 60%, by weight of isobornylacetate and 40%, by weight, of the condensation product of 1 mol of nonyl phenol and 10 mol of ethylene oxide),
0.1 parts, by weight, of water-soluble dye (C.I. No. 42085),
0.2 parts, by weight, of a bactericide (sodium salt of benzoic acid) and
0.2 parts, by weight, of sodium hydroxide.
- After 45 seconds stirring, a reaction sets in with mild foaming. The setting time is 50 seconds. After 10 minutes, the perfume carrier has a gross density of 650 kg/m³. The gel begins to shrink after several hours so that the gross density after 24 hours is 750 kg/m³.
- Example 2**
15 parts, by weight, of the prepolymer prepared according to 1(a), which still contains isocyanate end groups, are added to a solution of:
- 105 parts, by weight, of water,
10 parts, by weight, of the perfume oil from Example 1,
0.1 parts, by weight, of the dye from Example 1, and
0.2 parts, by weight, of the bactericide from Example 1
- with vigorous stirring (speed of stirrer 1200 revs/min). A gel begins to form after the mixture has been stirred for 80 seconds. The perfume carrier obtained has a foamy character. After 24 hours, it has a gross density of 750 kg/m³.
- Example 3**
16.5 parts, by weight, of the prepolymer described under 1(a) are added to a solution of:
- 62 parts, by weight, of water,
58 parts, by weight, of propan-2-ol,
10 parts, by weight, of the perfume oil from Example 1,
- 0.1 parts, by weight, of the dye from Example 1,
0.2 parts, by weight, of the bactericide from Example 1 and
0.2 parts, by weight, of sodium hydroxide.
- 16.5 parts, by weight, of the prepolymer des-
- 0.1 parts, by weight, of the dye from Example 1,
0.2 parts, by weight, of the bactericide from Example 1 and
0.2 parts, by weight, of sodium hydroxide,
- using a stirrer rotating at 1200 revs/min. A gel begins to form after the mixture has been stirred for 120 seconds, and the solidified perfume carrier is obtained after a further 10 seconds.
- Example 4**
A solution of:
- 90 parts, by weight, of water,
0.1 parts, by weight, of 1,2 - diaminoethane,
0.2 parts, by weight, of the dye from Example 1,
0.4 parts, by weight, of the bactericide from Example 1 and
11 parts, by weight, of the perfume oil from Example 1
- are rapidly added to a dispersion of
- 15 parts, by weight, of the prepolymer described under 1(a),
100 parts, by weight, of water and
20 parts, by weight, of titanium dioxide powder
- with stirring (at 1200 revs/min). The perfume carrier is obtained in the form of a gel after 4 seconds.
- Example 5**
(a) Preparation of the prepolymer:
386 g of a polyether having a hydroxyl number of 28 based on glycerol and containing 60 percent, by weight, of ethylene oxide and 40 percent, by weight, of propylene oxide are introduced into a reaction vessel and 114 g of 1,6 - diisocyanatohexane are added with stirring. The mixture is heated to 120°C within 30 minutes with stirring and then maintained at this temperature for a further 4 hours. The prepolymer has an isocyanate content of 10.5%, by weight, and a viscosity of 1440 centipoises at 25°C.
- (b) Preparation of the perfume carrier:
A solution is prepared from:
- 115 parts, by weight, of water,
8 parts, by weight, of the perfume oil from Example 1,
0.1 parts, by weight, of the dye from Example 1,
0.2 parts, by weight, of the bactericide from Example 1 and
0.2 parts, by weight, of sodium hydroxide.

cribed under (a) are added to this solution with vigorous stirring. When the reaction mixture has been stirred for 75 seconds, it foams slightly and begins to change into a gel-like state. The gross density of the perfume carrier obtained is 650 kg/m³ after 24 hours.

Example 6

A solution, consisting of:

- 65 parts, by weight, of water,
- 10 parts, by weight, of the perfume oil of Example 1,
- 0.1 parts, by weight, of the dye from Example 1,
- 0.2 parts, by weight, of the bactericide from Example 1,
- 0.45 parts, by weight, of 1,2 - diaminoethane and
- 0.7 parts, by weight, of a 43% solution of the sodium salt of N - (2 - aminoethyl) - 2 - amino - ethane sulphonic acid

is added with stirring (1000 revs/min) to an emulsion of 8.2 parts, by weight, of the prepolymer described under 5(a) in 50 parts by weight of water. Gel formation sets in after a stirring time of 20 seconds. The perfume carrier obtained has a gross density of 870 kg/m³.

Example 7

A solution is prepared from:

- 100 parts, by weight, of water,
- 1 part, by weight, of 1,2 - diaminoethane,
- 0.1 parts, by weight, of the dye from Example 1,
- 0.4 parts, by weight, of the bactericide from Example 1 and
- 11 parts, by weight, of the perfume oil from Example 1.

This solution is added with stirring to a dispersion of

- 15 parts, by weight, of the prepolymer described under 5(a),
- 50 parts, by weight, of water and
- 20 parts, by weight, of titanium dioxide powder.

A gel forms after 4 seconds' stirring. The perfume carrier has a gross density of 870 kg/m³.

Example 8

- 15 parts, by weight, of the prepolymer described under 5(a) are emulsified in
- 50 parts, by weight, of water. A solution of:

- 100 parts, by weight, of water,
- 1 part, by weight, of 1,2 - diaminoethane,
- 0.1 parts, by weight, of the dye from Example 1,
- 0.4 parts, by weight, of the bactericide from Example 1 and
- 11 parts, by weight, of the perfume oil from Example 1

is then added. The reaction sets in after a stirring time of 4 seconds.

Being aware of the Carcinogenic Substances Regulations, 1967, we make no claim herein to the use of benzidine in contravention of these Regulations.

WHAT WE CLAIM IS:—

1. An odourant composition comprising, as carrier, a polyurethane - polyurea polymer gel containing, as dispersing agent, water and/or an alcohol and an ethereal oil, perfume or scent which is soluble, emulsifiable or dispersible in the dispersing agent.
2. A composition as claimed in claim 1 substantially as herein described.
3. A composition as claimed in claim 1 substantially as herein described with reference to any one of the Examples.
4. A process for the preparation of a composition as claimed in claim 1 which comprises reacting, in a reaction medium of water and/or an alcohol, an isocyanate group-containing prepolymer, which has been prepared from a polyether containing at least 40% by weight, of ethylene oxide units, with, as chain-lengthening agent, water or a di- or higher poly-amine in the presence of an ethereal oil, perfume or scent which is soluble, emulsifiable or dispersible in the reaction medium.
5. A process as claimed in claim 4 in which the said prepolymer is prepared using an NCO:OH ratio of from 1.5 to 20.
6. A process as claimed in claim 5 in which the said ratio is from 2 to 10.
7. A process as claimed in any of claims 4 to 6 in which the said prepolymer has an isocyanate content of from 2 to 20%, by weight.
8. A process as claimed in claim 7 in which the said isocyanate content is from 5 to 10%, by weight.
9. A process as claimed in any of claims 4 to 8 in which the said polyether has a molecular weight of from 500 to 10,000.
10. A process as claimed in claim 9 in which the said molecular weight is from 2000 to 8000.
11. A process as claimed in any of claims 4 to 10 in which the said polyether contains at least 50%, by weight, of ethylene oxide units.
12. A process as claimed in claim 4 substantially as herein described.

13. A process as claimed in claim 4 substantially as herein described with reference to any one of the Examples.

- 5 14. An odourant composition as claimed in claim 1 when prepared by a process as claimed in any of claims 4 to 13.

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